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NEWARK PLANT
PIGMENT COLOR RESEARCH REPORT
Final Report

CONTROL OF DUSTING OF "HY-CAP" PIGMENTS
GRANULAR MILORI BLUE B-278-D

Period Covered

1944 - 1950

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NEWARK PLANT

PIGMENT COLOR RESEARCH REPORT

Final Report

CONTROL OF DUSTING OF "HY-CAP" PIGMENTS
GRANULAR MILORI BLUE B-278-D

1944 - 1950

CHARGE: A-II-C-214
A-I-C-5

SUBMITTED BY: J. O. Morrison

DATE SUBMITTED: 1/12/51.

APPROVED BY: *B. H. Perkins* 2/9/51
B. H. Perkins

DATE ISSUED: 2/9/51

INTRODUCTION:

This report covers study directed toward development of economical methods for reducing the dusting of colored pigments, particularly "Hy-Cap" iron blues and chrome greens, conducted under Project A-II-C-214, and application of these principles to the development and standardization of B-278-D.

SUMMARY AND CONCLUSIONS:

Study of the principles involved in controlling the dusting of these pigments has been completed. An improvement in dusting behavior is effected by micro-pulverizing the lump pigment coarsely, at the same time treating the coarsely ground powder with a high-boiling petroleum-solvent, and subsequently pelletizing the treated pigment by mixing it in a standard color blending mixer such as is normally used in the regular bag packing operation.

Development study has included preparation of sizable quantities of the desired reduced dusting super-texture iron blue, and chrome greens at four standard shades of the G-550-D line.

Laboratory testing methods have been developed for the evaluation of dusting tendencies and roller mill grinding properties (freedom from separation and tailings). The results obtained with these testing procedures have been corroborated by practical evaluation of a few of the semi-works products.

Experimental evidence indicates that the incorporation of the anti-dusting oily agent is an essential step in the proposed procedure. The preferred procedure has been cleared by the Legal Department as free from domination by the Cabot "spheronizing" patents.

To date the experimental process has been applied only to iron blue. A product suitable only for ball mill use, comprising treated B-216-D type pigment and coded B-278-D, is regularly manufactured and sold to Intag. The attached B-278-D(A) making and processing instructions and notes describe the process completely.

The softer texture product required for satisfactory roller mill grinding has not been manufactured. Release of any such product for roller mill application must be governed by adequate large scale pretesting to provide assurance of satisfactory roller mill performance.

PATENT SITUATION:

The preferred procedure effecting the improvement in dusting behavior has been cleared by the Legal Department as free from domination by the Cabot "Spheronizing" patents. It appears that we are free to operate our process.

REFERENCES: DSN-45-8 and 51-3.
Major Experimental Project No.214
Notebook 1075.

Cost Group - 5

(3135)

Code: B-278-D

K# (A)

Approved by:

Goal: 4292#

JOM: 12-5-49

Diazo Vats

HA: 12-12-49

Coupling Vat:

CWO: 12-15-49

Strike Vat: 181, 280, 282

JES: 3-2-50

Std. Hours 6.0 Non-Bleeding

VAT	NAME: MILORI BLUE	INSTRUCTIONS	USE LBS.	N CODE	ACCT. LBS.
380	1. Pump into scale tank 380				
1	Alk. _____ % CNxs _____ T _____ M _____ %			594	5897
	Alk. _____ % CNxs _____ T _____ M _____ %			594	
	A. Start water into scale tank 380.				
	B. Turn on agitator and steam.				
	C. Weigh and dump in			4	
	D. Adjust to 77" (3053G.) at 104-106°F.				
	E. Stir 20 minutes or longer to dissolve.				
Misc.	2. Weigh and hold at vat 383 for step 5		1700	340	1700
2					
383	3. Run 15-20" (+245G.) water into vat 383				
3	4. Turn on agitator and steam.				
	5. Dump in N-340 weighed in step 2.				
	6. Adjust to 40" (490G.) at 70-75°F.				
	7. Stir 20 minutes or longer to dissolve.				
S.V.	8. Remove steam vent seal in strike vat.				
4	9. Get inside strike vat and hose top, wall, agitator etc. thoroughly clean.				
	10. Run 20-25" (+2625G.) water into strike vat with agitation.				
	11. Blow steam lines.				
	12. Run wash water to sewer with agitation.				
	13. Set agitator at 16 RPM.				
389	14. Weigh and dump into vat 389.		226	12	226
5	15. Adjust to 24" (40G.).		226	12	226
	16. Heat to 120-140°F to dissolve.				
387	17. Run into scale tank 387 from vat 343				
6	M _____ T _____ %			8	71
	18. Adjust to 24" (40G.).				
	19. Stir to uniform solution.				
388	20. Weigh and pour into vat 388		243	399	243
7	21. Start agitator.				
	22. Adjust to 30" (86G.) by adding water slowly.				
	23. Stir 3-5 minutes to emulsify.				
	24. Weigh and pour in 2-3 minutes		65	122	65
	25. Stir 5 minutes.				
	26. Check and Record: Solution _____, must be dark and clear.				
S.V.	27. When strike vat runs empty hose top, wall, agitator, etc. from outside of vat and run to sewer.				
	28. Run 5-15" (+1050G.) water into strike vat.				

VAT	PAGE: 2	CODE: B-278-D(A)	Lbs: 4292#	USE LBS.	N CODE	ACCT. LBS.
		29. Pump into strike vat from Vat 382				
		pH _____ Fe _____ M _____ %			24	3894
		pH _____ Fe _____ M _____ %			24	
		30. When step 7 stirring completed, run vat 383 into strike vat through screen.				
		31. Install chart in temperature controller.				
		32. Adjust to 39" (4095G.) at 74-76°F.				
380		33. Adjust scale tank 380 to 77" (3053G.) at 104-106°F				
9						
S.V.		34. Strike vat 380 at 104-106°F into strike vat at 74-76°F. on the surface in 40-45 min.				
10		35. Run in from scale tank in 5-10 minutes.				
		M _____ T _____ %			27	2349
		36. If N-594 alkalinity in step 1 exceeds 0.35% calculate and run into strike vat from scale tank.				
		(%Alk-0.35) = _____ %x(use lbs N-594) ÷ 100 =				
		acct. lbs. N-27			27	
		37. Stir 5 minutes.				
		38. Check and Record: _____ °F.				
		39. Test and Record: Copperas XS _____, Must be +.				
		40. Heat at once to 169-171°F in 57-63 minutes.				
		Record: Time started _____.				
		41. Check and Record accuracy of controller:				
		Low: Thermometer _____ °F Controller _____ °F.				
		High: Thermometer _____ °F Controller _____ °F.				
		42. Stir 30 minutes at 169-171°F.				
		Record: Time started _____.				
		43. Run vat 389 into strike vat in 2-4 minutes.				
		44. Stir 15 minutes.				
		45. Run vat 387 into strike vat in 2-4 minutes.				
		46. Stir 15 minutes.				
		47. Run vat 388 into strike vat.				
		48. Stir 2 hours. Record: Time Started _____.				
		49. Record: pH _____. Normal 1.5-2.2. If above pH 2.5 Adjust downward only to 2.0-2.5 with N-27. Do not make upward adjustment. Record: pH _____.				
		50. Shut off agitator.				
		51. Air cool for 8 hours or less to 140-145°F.				
		Record: Time started _____.				
		Record: Time stopped _____.				
		Record: _____ °F.				
		52. 3 hours after start of air cooling, turn on agitator for 3-5 minutes.				
		53. 6 hours after start of air cooling, turn on agitator for 3-5 minutes.				
		54. Before recording final temperature in step 51, Stir 3-5 minutes.				
		55. Check and record: _____", Normal 77"(8085G.).				
		56. If necessary, flood to 140-145°F.				
		Record: _____ °F.				
		Record: _____".				
		57. Press with agitation.				
		GRIND WITH				
		" "				
		(440 Cu.Ft.)				
			0	621	172	
			0	622	440	
		TIME START _____ TIME FIN. _____ OPTR. _____				
		" " " " " "				

Code: B-278-D 4292# or * Grind
Goal: (4120# or * Lump)
Process: (A) Date: HA: 12-12-49

MAKING AREA NOTES JES: 3-2-50

PRESSING AREA INSTRUCTIONS

PRESSING AREA NOTES

Vat
Date
Time
Released by
Press No. 32,33,36,37,38,39) for each
No. of Cakes 60,60 60 60 60 60) 1/3 of batch
Press with Agitation
Wash to 20 hours or 1800 ohms.
Make first test after 15 hrs.
Load on 7 cars for each 1/3 of batch.
Special Instructions Non-Bleeding.
Wash with 100-140°F water.
Leave top 4 pans empty on each car.
Samples
Deliver to

DRYING AREA INSTRUCTIONS

DRYING AREA NOTES

Type L.V. H.V. Hurricane
Hours 10 + 9 ---- 10 + 9
Temp. 200 = 140 ---- 200 140

Lump container See Chart.
Special instructions Non-Bleeding

Samples
Deliver to

GRINDING AREA INSTRUCTIONS

GRINDING AREA NOTES

Grinder No. 3 L.S.P. 5/8" screen
Special Instructions Non-Bleeding.
Follow special directions on back
of card.
Samples: 2 envelopes & 1 Qt. Jar 1/2 full.

Deliver to: Control Laboratory
Mix before sampling 60 Min.
No. 1 Conical Blender.
Packaging: See chart.

2nd. Split entirely consumed, per
scheduling table.

SPECIAL GRINDING INSTRUCTIONS

See Job Breakdown for Details.

1. Check mill and N-621 pump speed (180 RPM).
Adjust spray to 40-50# oil pressure.
2. Fill N-621 supply tank.
3. Crush lump and load in hopper.
4. When starting grind, ALWAYS:
 - A. Turn on 8 lbs. Nitrogen
 - B. Start Grinding
 - C. Open N-621 Valve.
 - D. Start N-621 Pump.
5. Record accurate time _____ Starting 1st Drum.
6. Immediately check spray from gun.
7. When stopping grind, ALWAYS:
 - A. Stop N-621 Pump.
 - B. Close N-621 Valve.
 - C. Stop Mill.
 - D. Turn Nitrogen valve LEFT.
8. Frequently inspect color for uniform appearance.
9. Frequently inspect spray from gun.
10. Record accurate time first drum is filled _____
11. Continue grinding into second drum.
12. Record net weight of first drum _____#.
13. Divide lbs. _____ + _____ minutes = _____# per min.
If less than 8 or more than 10 multiply by 19.2 and correct N-621 pump speed.
14. Continue grinding batch, per instructions 4,7,8,9.
15. Open and clean mill whenever sludge appears in product.
16. Weigh total batch. Record: _____# Grind.
17. Subtract: _____# Lump.
= Lbs. N-621

MIXING INSTRUCTIONS

- A. Divide middle batch of 3 batch family(1 strike) to obtain 2 equal mixes from the 3 batches.
- B. Record following on CARD FOR 1ST.BATCH ONLY:

1st.Lot# _____, _____ Drums, + _____ Drums from 2nd.
2nd.Lot# _____, _____ Drums, Divide between 1st and 3rd
3rd.Lot# _____, _____ Drums, + _____ Drums from 2nd.
Total Drums _____ + 2= _____ Drums each mix.

- C. Tumble each mix 60 minutes. Inspect dusting.
Continue tumbling if necessary until satisfactory.
- D. Submit samples to Lsb.
- E. Hold in blender for disposition.
- F. When OK, pack in 50 lb. bags.
Handle carefully to avoid breaking granules.

Newark, New Jersey
December 15, 1949

B-278-D(A)
STANDARD FORMULA NOTES

B-278-D is a green toned Milori Blue in dustless, free-flowing granular form. Lump pigment is manufactured by the B-271-D "B" process, then coarsely pulverized and simultaneously treated, in the pulverizer, with 4% of high-boiling hydrocarbon oil (Mentor 28), and finally converted to the granular form by tumbling in a conical mix-tank.

Practical evaluations conducted during the Chemical Division process development study (Project 214) demonstrated that, while B-278-D as manufactured by the "A" process can be employed in ball-mill dispersion applications with considerable advantage because of its freedom from the objectionable dusting which characterizes the use of conventional powdered pigments, its use in roller-mill dispersion operations cannot be generally recommended because of adverse tendencies toward particle-classification and pigment separation on the roller-mill, resulting from incomplete wetting of the pigment with vehicle in the pregrind mixing process. Although the latter difficulties are greatly minimized by use of the experimental (B) B-278-D process (inert atmosphere strike), the "B" has not been standardized to date because of cost considerations.

At the insistence of the Intag Division of Interchemical Corporation, which cooperated in practical ball-mill evaluation phases of the development study, B-278-D(A) has recently been standardized and released. Sale of the pigment is temporarily restricted to Intag.

MAKING:

The making process is identical with B-271-D "B".

All stepwise making instructions must be followed explicitly. Particularly critical are the weights of N-594 (Step 1) and N-340 (Step 2), and the time and temperature specifications in Steps 40 and 42.

The process responds tinctorially to normal iron blue control variables. It should not be necessary to use any variable other than weight of N-340, with suggested limits of 1600 and 1875 lbs.

In the unexpected event that the process should "go" out of control it would be desirable to institute lump rubouts (96% lump, 4% N-621, versus "as is" standard) as a screening test for diversion of unsatisfactory batches to other channels, such as B-216-D, prior to grinding.

ALTERNATE PRODUCTION:

When desired, lump color for conversion to B-278-D may be selected from existing stock of B-271-D on the basis of rubout of a representative sample of the B-271-D, in which 4% of the standard rubout weight of the lump sample is replaced with N-621.

PRESSING AND WASHING:

In conformance with current operating practice the parent batch is pressed in three wash presses under separate lot numbers. Maintenance of standard texture requires uniform and thorough washing.

DRYING:

Assured maintenance of fine quality, particularly texture, demands that no portion of the lump pigment be overdried. This requires that:-

1. Dryer pans must be uniformly loaded.
2. Deformed pans, which prevent air circulation, must not be used.
3. The specified high-temperature drying period must never be exceeded by the Drying Room.

The specified drying cycle, 10 hours at 200°F. followed by 9 hours at 140°F., produced uniformly excellent results, during winter months, with B-269-D, a very similar product but with unusually exacting texture requirements. The cycle may require seasonal change to avoid excessive moisture during the summer. All such changes must be dominated by the first paragraph in this section. The writer's experience has indicated that the high-temperature period should probably never exceed 14 hours.

GRINDING AND MIXING:

A. General:

A relatively coarse grind is essential since the quantity of N-621 (Mentor 28 oil), required for subsequent elimination of the dust formed during grinding, must be kept at a minimum to avoid excessive dilution of the pigment tinting strength. Pulverizer No. 3, with 5/8" round perforated screen, must be used as specified.

The N-621 is continuously injected into the pulverizer rotor chamber, while grinding, by means of a spray gun to which the oil is metered through a variable-speed gear pump. The specified pump speed (192 RPM) provides the desired 4% treatment for the color grinding rate experienced to date but must be adjusted to compensate for any change in grinding rate, as detailed below.

The three 1431 lb. sister-batches of ground color obtained from a single 4292 lb. strike are combined into two 2146 lb. lots for the final granulation and dust removal operation in conical mixer No. 1. The specified 60 minute tumbling period has been adequate in the majority of batches to date. In the two unsatisfactory cases, an additional 90 minute tumbling period was arbitrarily employed with fully satisfactory results.

B. Safety:

No tendency toward increased inflammability as a result of Mentor treatment was detected in a careful examination of semi-works B-278-D treated with 7% of the agent.

At the suggestion of the Safety and Fire Protection Division of the Service Department, compressed nitrogen is employed as the Mentor atomizing medium. It is recognized that the quantity of nitrogen employed is much less than would be needed to maintain an inert atmosphere inside the pulverizer.

SAFETY PRECAUTIONS SPECIFIED FOR BLENDING OF IRON BLUE IN SEALED BLENDERS MUST BE OBSERVED.

"Always Be Careful".

C. Equipment Preparation:

1. Check interior of #3 mill. If necessary, remove sludge or install 5/8" RP Screen.
2. Check N-621 supply. Three batches (4292#) require 25 gallons.
3. Check N-622 supply. Three batches require 2 cylinders (440 cu.ft.).

4. With spray gun removed from mill and directed into a bucket,
 - A. Set N-622 pressure at 8 lbs.
 - B. Clamp gun trigger open, start and adjust N-621 pump to 192 RPM*, then adjust gun trigger to provide _____ lbs. N-621 line pressure.
 - C. If spray is not satisfactory, have gun cleaned by qualified expert.
 - D. When spray has been satisfactorily adjusted, do not disturb trigger adjustment, but shut off N-621 supply, then shut off N-622 gas supply.
5. Install spray gun on supporting rods on side of #3 mill with nozzle 3/4" outside of open mill port.
6. Fill N-621 supply can to top.

Notes:

- a). The N-621 oil reservoir calibration is 3.86 lbs. N-621 per inch.
- b). The gear pump assembly is equipped with two pumps. The smaller one has been used exclusively to date. The larger one will be needed in the future as noted under (e) below.
- *c). At 192 RPM the small pump will deliver 24 lbs. of N-621 per hour, equivalent to 4% treatment at a grinding rate of 600 lbs. per hour. Actual grinding rates have varied between 500 and 600 lbs. per hour. Experience has indicated that the 192 RPM rate may be safely used between these limits, insofar as quality is concerned. Because of the low cost of N-621 and the minor effects of such variations on total pigment yield, there is no need for accurate batchwise revision of the standard 4% treatment quantities as charged out on the individual making cards; periodic adjustment of N-621 (as well as N-622 Nitrogen) accounts can be made, based on actual inventory.
- d). Daily spot checks of actual grinding rate (accurate time for one barrel) are recommended, with appropriate adjustment of N-621 pump speed if grinding rate is slower than 500 or faster than 600 lbs. per hour. The following formula applies for calculating correct speeds for the small pump:

$$\text{RPM for 4\%} = \frac{192}{600} \times 60 \times \frac{\text{lbs. ground}}{\text{minutes time}} = 19.2 \times \frac{\text{lbs.}}{\text{mins.}}$$

- e). It is expected that projected replacement of the lump color feed hopper will increase the grinding rate to approximately 1000 lbs. per hour, which was regularly obtained with a similar semi-works mill. This will necessitate use of the larger pump and determination of the correct RPM to deliver 4% N-621 based on actual grinding rate.

D. Grinding:

1. To obtain satisfactorily uniform grinding rate and uniform N-621 treatment, it is necessary to thoroughly crush each drum of lump color before dumping 1/2 of contents into feed hopper. Repeat crushing before dumping last 1/2 of contents.
2. ALWAYS observe following sequence when starting to grind:
 - A. Turn on nitrogen supply and adjust to 8 lbs. pressure.
 - B. Start mill.
 - C. When color is discharging, but NOT BEFORE, open N-621 valve and start N-621 pump.
3. Immediately check to be sure spray is functioning properly.
4. Check constantly to be sure color continues to grind at normal rate.
5. Whenever mill is stopped for ANY reason, ALWAYS observe following sequence:
 - A. Shut off N-621 pump and close N-621 valve.
 - B. Let mill run 1 to 2 minutes (except in emergency), then stop mill.
 - C. Stop nitrogen flow. (TURN GAUGE VALVE TO LEFT.)
6. Check each drum of ground color for uniform appearance. If oily sludge is present in color, the mill must be opened and cleaned.
7. Periodically check appearance of spray while grinding. Keep port hole open and clear, by locking out mill and scraping away accumulated sludge as necessary.
8. Open mill and remove sludge and cake, when necessitated either by presence of sludge in discharged color or by excessive squealing of drive pulley.
9. Record number of drums ground, see instruction 1 below.

E. Mixing:

Note: Record following data for each three-batch family on first batch card only.

1. When all three lots of a three-batch family (single strike) have been ground, record number of drums in each lot. Distribute ALL of 2nd lot between 1st and 3rd to obtain two equal size mixes. Record:

1st lot No. _____, _____ drums + _____ drums from 2nd lot.

2nd lot No. _____, _____ drums, to be divided between Lots 1 & 3.

3rd lot No. _____, _____ drums, + _____ drums from 2nd lot.

Total drums in 3 lots _____ $\div$ 2 = _____ drums for each mix.

2. Tumble each mix 60 minutes in No. 1 conical blender, sample, and inspect dusting. Continue tumbling until dusting appears satisfactory. Then submit laboratory samples and hold in blender for disposition.
3. When OK, pack and handle carefully to avoid breaking granules.

PACKAGING:

No evidence of oil-staining of bags was obtained in a 29 month storage test in the semi-works.

No information is available concerning degree of granule breakdown and dust formation resulting from transportation of the color, either in bags or in rigid containers. Bags should be used exclusively in the absence of specific requests to the contrary.

TESTING:

Routine control tests are limited to dust inspection and rubout evaluation.

The dust inspection consists of visual examination of a pint sample in a quart glass jar. No more than an extremely slight amount of suspended color should be visible immediately after the jar is moderately shaken.

Suggested rubout shipping limits are:

Masstone:	dark - s. light
Undertone(Trans.)	vs.green - vs.red
Strength:	95 - 105
Tint:	s.green - s.red
Tint:	vs.intense - vs.dull

Because of the nature of the product non-representative sampling for the rubout test is occasionally experienced. No batch should be rejected until substandard color results are adequately confirmed by new samples.

QUALITY PERFORMANCE:

Experience to date indicates that no reblending of granulated pigment should be necessary inasmuch as the color performance of the basic process (B-300-D, B-271-D) is remarkably consistent.

In the event that reblending should be required, it must be done in conical mixer #1. Ribbon mixers disintegrate the granules almost instantaneously.

YIELD PERFORMANCE:

Experience to date supports the assumption that all of the N-621 is retained in the product.

CALCULATED COMPOSITION (NB. 951-1.47)

Anhydrous Cyanoferrate Complex	3719 lbs.	86.65%
Organic Treating Agents	318 "	7.40
Moisture	255 "	5.95
	<u>4292 lbs.</u>	<u>100.00%</u>

CALCULATED GOAL YIELD (NB. 951-1.47)

Anhydrous Lump	-	3865 lbs.
As Is Lump (6.2% H ₂ O)	-	4120 "
Anhydrous Grind	-	4037 "
As Is Grind (5.95% H ₂ O)	-	4292 "

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RMK